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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 21926-2016

Determination of 2-dodecylcyclobutanone in irradiated foods containing fat -- GC/MS method

食品安全国家标准 含脂类辐照食品鉴定 2-十二烷基环丁酮的气相色谱-质谱分析法

(English Translation)

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Foreword

Instead of the standard GB/T 21926-2008 "irradiated food containing fat 2-dodecyl-azetidinone by Gas Chromatography/Mass Spectrometry." as compared with the present standard GB/T 21926-2008, the main changes are as follows.

--- standard name changed to "Food Safety Standard irradiated food containing lipid-2-dodecyl cyclobutanone identified by gas chromatography -Mass Spectrometry ";

--- Improved silica gel column chromatography.

National Food Safety Standard Identification of lipid-containing irradiated food 2-dodecyl cyclobutanone GC -Mass spectrometry

1 Scope

This standard specifies the use of 2-dodecyl-azetidinone lipid-containing food irradiation identified by gas chromatography - mass spectrometry.

This standard applies to the identification of irradiated food fat content greater than 1%.

Principle 2

After extraction using n-hexane fat sample cyclobutanone and 2-dodecyl out together, purification by silica gel column chromatography, concentrated gas Chromatography - mass spectrometer 2-dodecyl cyclobutanone, external standard.

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3 Reagents and materials

Unless otherwise indicated, the reagents used in this method are analytically pure water three water GB/T 6682 provisions.

3.1 Reagent

3.1.1
hexane (C6H14).

3.1.2
anhydrous sodium sulfate (Na2SO4). 650 °C burning 4h, cool in a desiccator, stored for use.

3.1.3
ether (C4H10O).

3.1.4
nitrogen (N2). purity >=99.999%.

3.1.5
helium (He). purity >=99.999%.

3.2 Material

Silicone. 250μm ~ 500μm, column chromatography. Before use, the activated silica gel placed in an oven at 100 deg.] C 12h, cool in a desiccator. in Stoppered Erlenmeyer flask was added 4% (mass ratio) of water deactivation of the conical flask shaken until uniformly dispersed silica. The deactivated silica gel in a desiccator Ping Heng 12h, available within a week.

3.3 Standard

2-dodecyl cyclobutanone standard (C16H30O). purity >=99%.

3.4 Standard Preparation

3.4.1 2-dodecyl azetidinone standard stock solution Accurately weighed amount of 2-dodecyl cyclobutanone standard (accurate to 0.1 mg), was dissolved with n-hexane, formulated at a concentration of 100μg/mL of Standard stock solution, -20 °C storage, 3 months effective.

3.4.2 2-dodecyl azetidinone standard working solution Standard stock solution was diluted with hexane (100μg/mL), were formulated as 0.10μg/mL, 0.25μg/mL, 0.50μg/mL, 1.00 Standard working solution levels μg/mL, 2.50μg/mL, 5.00μg/mL of, -20 °C storage 1 month

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effective, but appear abnormal peaks You should consider re-formulated.

4 Instruments and equipment

4.1
GC-MS. with a less polar (e.g., DB-5) and suitable properties or specifications of the capillary column, length. 30m, inner diameter. 0.25mm, film Thickness. 0.25μm.

4.2
Soxhlet extractor.

4.3
Nitrogen blowing instrument.

4.4
electric mixer.

4.5
Ovens.

4.6
temperature water bath.

4.7
rotary evaporator.

4.8
Electronic balance. a sense of the amount of 0.1mg.

4.9
Glass chromatography columns. column length.200mm ~ 300mm, inner diameter. 20mm.

b) inlet temperature. 250 °C;

c) Interface temperature. 280 °C;

d) Ion source. EI source, 70eV;

e) Measurement mode. SIM (the SIM); ion monitoring (m/z). 55,98,112, quantitative ion (m/z). 98;

f) Carrier gas. Helium, flow rate 1.0mL/min;

g) Injection volume. 1.0μL;

h) Injection mode. splitless.

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6 Expression analysis results

Standard working solution and the volume of the sample solution and the like is inserted into the sample measured parameters, external standard. In the chromatographic conditions, 2-dodecyl cyclobutanone Selected ion chromatogram of the standard sample shown in Figure A.1.2- dodecyl cyclobutanone retention time of approximately 17.1 min, mass spectrum is shown in Figure A.2.

2-dodecyl cyclobutanone peaks detected if the sample liquid retention time retain the 2-dodecyl cyclobutanone standard peaks Same time (relative deviation <=2s), which spectrum is consistent with the 2-dodecyl cyclobutanone standard spectrum, after deducting the background monitor select Measuring ions m/z55,98 and 112 both occur, and the relative abundance of m/z55,112 and the base peak m/z98 and the ratio of standard 2-dodecyl cyclobutanone Registration deviation relative abundance ratio not exceeding 20%, can be detected in the sample confirmed 2-dodecyl cyclobutanone, indicated the sample had received irradiated deal with.

Appendix A

2-dodecyl cyclobutanone standard chromatogram and mass spectrum A.1 2-azetidinone-dodecyl selected ion chromatogram of the standard 2-dodecyl azetidinone standards selected ion chromatogram shown in Figure A.1.

FIG A.1 2- azetidinone-dodecyl selected ion chromatogram of the standard

A.2 2- azetidinone-dodecyl standard full scan mass spectrum 2-dodecyl azetidinone standard full-scan mass spectra shown in Figure A.2.

FIG A.2 2- azetidinone-dodecyl standard full scan mass spectrum

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